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(54) HEAT RESISTANT MAGNESIUM ALLOY MOLDED MEMBER, HEAT RESISTANT MAGNESIUM ALLOY USED FOR THE MOLDING AND MOLDING METHOD THEREFOR

(57)Abstract:

PROBLEM TO BE SOLVED: To produce a heat resistant Mg allay molded member excellent in moldability and extensibility while its creep resistance is secured, in an Mg-Al-Ca-Mn alloy, by specifying its compsn. and regulating the ratio of Ca/Al to a specified value or below.

SOLUTION: An Mg alloy having a compsn. contg., by weight, 2 to 6% Al, 0.5 to 4% Ca, and the balance Mg, and in which the ratio of Ca/Al is regulated to ≤0.8 is subjected to half-melting injection molding at a liquids temp. or below in which solid phases and liq. phases are coexistent. In this way, the heat resistant Mg alloy molded member such as automobile transmission parts or engine parts excellent in creep resistance can be obtd. Al is required by ≥2%, but, in the case of >6%, its elongation deteriorates even if the half-melting injection molding is executed. Ca is added for increasing its high temp. strength tending toward reduction accompanying the addition of Mg to Al, but, for preventing the reduction of its moldability and the elongation of the molded member, the ratio of Ca/Al is required to be suppressed to ≤0.8. Moreover, ≤0.15% Sr is preferably added as a refining agent.

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CLAIMS

[Claim(s)]
[Claim 1] The heat-resistant Magnesium alloy shaping member which is a shaping member by which 2 - 6 % of the weight of aluminum and 0.5 - 4 % of the weight of calcium were contained, the remainder consisted of magnesium and an unescapable impurity, and the calcium/aluminum ratio was fabricated from 0.8 or less Magnesium alloy, and is excellent in creep resistance.
[Claim 2] The heat-resistant Magnesium alloy shaping member according to claim 1 in which a calcium/aluminum ratio has the creep resistance below the test temperature of 150 degrees C, and minimum creep rate 4x10-10/S in test load 50MPa or less by 0.6.
[Claim 3] The heat-resistant Magnesium alloy shaping member according to claim 1 or 2 in which a Magnesium alloy contains 0.15 more or less % of the weight of Sr.
[Claim 4] The heat-resistant Magnesium alloy shaping member according to claim 1 to 3 whose diameter of average crystal grain is 30 micrometers or less.
[Claim 5] The heat-resistant Magnesium alloy shaping member according to claim 1 to 4 whose shaping components are the transmission components for automobiles, or engine components.
[Claim 6] The heat-resistant Magnesium alloy with which the creep resistance which was excellent with half-melting injection molding in the temperature below the liquidus line with which 2 - 6 % of the weight of aluminum and 0.5 - 4 % of the weight of calcium are contained, the remainder consists of magnesium and an unescapable impurity, and solid phase and the liquid phase are intermingled is acquired.
[Claim 7] The heat-resistant Magnesium alloy according to claim 6 with which a Magnesium alloy contains 0.15 more or less % of the weight of Sr.
[Claim 8] The heat-resistant Magnesium alloy according to claim 6 or 7 whose calcium/aluminum ratio is 0.8 or less.
[Claim 9] The heat-resistant Magnesium alloy according to claim 8 whose calcium/aluminum ratio is 0.6 or less.
[Claim 10] The heat-resistant Magnesium alloy according to claim 6 to 9 which is the metal grain or pellet gestalt which introduced internal distortion.
[Claim 11] The shaping approach of a heat-resistant Magnesium alloy shaping member of excelling in the creep resistance characterized by carrying out half-melting injection molding of the Magnesium alloy according to claim 6 to 10 below at the liquidus-line temperature to which solid phase and the liquid phase are intermingled.
[Claim 12] The shaping approach of a heat-resistant Magnesium alloy member according to claim 11 that the rate of solid phase at the time of a half-melting condition is 30% or less in case half-melting injection molding is performed.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the heat-resistant Magnesium alloy shaping member which is excellent in especially a moldability and extensibility, the heat-resistant Magnesium alloy used for the shaping, and its shaping approach, securing creep resistance.
[0002]
[Description of the Prior Art] Also in the metallic material by which current utilization is carried out, a Magnesium alloy is a low consistency most and is strongly expected as future lighter weight materials for automobiles. The Magnesium alloy present most generally used is a Mg-aluminum-Zn-Mn system alloy (for example, AZ91D alloy), its room temperature reinforcement is high, and since corrosion resistance is also good, it is applied to the gearbox casing for automobiles, the cylinder head cover, etc. However, in the temperature region exceeding 120 degrees C, a strength property begins to fall and there is a fault that especially creep resistance is inferior, and on real part article level, the bearing surface of the screw conclusion section passes and it leads to the problem of slack etc.
[0003] On the other hand, as an alloy which has improved thermal resistance, AS41 Magnesium alloy of a Mg-aluminum-Si system is used. however -- being heavy-gage for becoming a property with about 150 degrees C insufficient [service temperature], although it is better than the above-mentioned AZ91D, and moreover, securing demand reinforcement about creep resistance, since a room temperature and an elevated temperature have the low tensile strength property -- not carrying out -- it does not obtain but the problem that the lightweight-ized effectiveness of magnesium decreases arises.
[0004] In addition, although there are alloys, such as QE22 which added silver, rare earth elements, etc. as an alloy which has improved thermal resistance, it is expensive and dies casting has a not suitable fault in respect of fluidity.
[0005] Therefore, the Mg-aluminum-calcium-Mn system alloy (JP,6-25790,A) which is newly excellent in high temperature strength came to be proposed. It is supposed that the high-temperature-strength property which 0.7 and the organization gestalt of the sludge which will be crystallized in a Magnesium alloy if it carries out to 0.75 or more preferably changed, and the Mg-calcium compound crystallized the ratio of calcium/aluminum, and was excellent comes to be shown especially here.
[0006]
[Problem(s) to be Solved by the Invention] However, it was easy to generate hot tearing, and when it is going to fabricate a member in dies casting in the high Magnesium alloy of a calcium/aluminum ratio, if molten metal temperature is high, with it, it has left the problem of being easy to generate the seizure to metal mold. This invention sets it as the 1st object to offer the heat-resistant Magnesium alloy shaping member which is excellent in a moldability and extensibility, securing the physical properties suitable for engine components, such as an automobile, etc., especially creep resistance in view of the technical problem which such a conventional technique has. The 2nd object of this invention is replaced with the dies casting used widely, and it is in offering the suitable shaping approach of the above-mentioned heat-resistant Magnesium alloy shaping member. Furthermore, the 3rd object of this invention is to offer the alloy presentation suitable for manufacturing the heat-resistant Magnesium alloy shaping member which is excellent in a moldability and extensibility,

securing the above-mentioned creep resistance.
[0007]
[Means for Solving the Problem] If the half-melting fabricating method for performing injection molding where it replaced with pressure die casting in the aluminum-calcium system Magnesium alloy and solid phase and the liquid phase are intermingled is application as a result of repeating examination variously, in order that this invention persons may solve the above-mentioned technical problem While printing of metal mold could be prevented, it found out that the reinforcement excellent in the member fabricated could be given, but in order to maintain the condition that the solid phase and liquid phase were intermingled, it is necessary to increase the addition of aluminum as much as possible. On the other hand, it adds in order for aluminum to dissolve with magnesium, to show age-hardening nature and to raise the mechanical property of an alloy, but in order to reinforce the high temperature strength in the inclination to fall, with addition of the aluminum to magnesium, adding calcium so that a calcium/aluminum ratio may be held or more to 0.7 is recommended (JP,6-25790,A). However, in order that a Mg-calcium system compound might crystallize so much when it is easy to generate printing to a casting crack and metal mold at the time of shaping if there are many these amounts of calcium, there is an inclination for the elongation of a moldings to fall and it found out that it was necessary to make a calcium/aluminum ratio or less into 0.8 rather.
[0008] Then, based on above-mentioned both knowledge, 2 - 6 % of the weight of aluminum and 0.5 - 4 % of the weight of calcium are contained, the remainder consists of magnesium and an unescapable impurity, this invention is the shaping member by which the calcium/aluminum ratio was fabricated from 0.8 or less Magnesium alloy, and it is to offer the Magnesium alloy shaping components which are excellent in a moldability and extensibility, securing creep resistance. Generally, in the Magnesium alloy, it dissolves with magnesium and age-hardening nature is shown, and in order to raise the mechanical property of an alloy, it is supposed that it is desirable to add 2 - 10 % of the weight of aluminum. On the other hand, although aluminum needs to be added 2% of the weight or more in this invention, if 6 % of the weight is exceeded, even if it performs half-melting injection molding, elongation will fall. Therefore, in order to attain expected effectiveness, performing half-melting injection molding, it is restricted to 6 or less % of the weight. On the other hand, although it is added in order to reinforce the high temperature strength in the inclination to fall, with addition of the aluminum to magnesium, calcium needs to press down a calcium/aluminum ratio or less to 0.8 so that elongation of a moldability and a shaping member may not be fallen, and, moreover, is restricted to 0.5 - 4% of the weight.
[0009] Although strontium is used as a detailed-ized agent in casting of magnesium, since the detailed-ized effectiveness of solid phase can be demonstrated also in the half-melting injection-molding method concerning this invention, adding is desirable. 0.15 or less % of the weight is suitable for an addition.
[0010] The above-mentioned shaping components have a diameter of crystal grain by 30 micrometers or less more than tensile strength 180MPa (298 degrees K: refer to drawing 9), and the outstanding creep resistance which is moreover the test temperature of 150 degrees C and below minimum creep rate 4x10-10/S in test load 50MPa is shown (based on the creep-under-tensile-force test method of a JISZ 2271"metallic material"). Therefore, it is suitable for the transmission components for automobiles, or engine components. Especially when a calcium/aluminum ratio is 0.6 or less, it excels in creep resistance.
[0011] moreover, in this invention, as an alloy raw material used for shaping of the above-mentioned Magnesium alloy shaping components 2 - 6 % of the weight of aluminum, and 0.5 - 4 % of the weight of calcium -- containing -- an impurity with the as unescapable remainder as magnesium, if it comes to contain 0.15 more or less % of the weight of Sr and it is required preferably A calcium/aluminum ratio also tends to offer the heat-resistant Magnesium alloy which is excellent in a moldability and extensibility, a calcium/aluminum ratio securing preferably the creep resistance which was excellent with half-melting injection molding adjusted to 0.6 or less 0.8 or less.
[0012] Especially as an alloy raw material, when fabricating by the half-melting injection-molding method, it is found out that it is effective in the formation of crystal detailed that it is the metal grain or pellet gestalt which introduced internal distortion (refer to drawing 10). As the processing

method, cutting is advantageous in cost.
[0013] Furthermore, it replaces with pressure die casting, and when considering the half-melting injection-molding method for performing injection molding where solid phase and the liquid phase are intermingled as application, it can carry out at the temperature below the low-temperature liquidus line from pressure die casting. Therefore, the shaping approach of a heat-resistant Magnesium alloy member of excelling in a moldability and extensibility is also offered, securing the creep resistance characterized by this invention carrying out half-melting shaping of the Magnesium alloy of the above-mentioned publication in the condition that the solid phase below liquidus-line temperature and the liquid phase are intermingled.
[0014] In order to inject dies casting at the temperature below the liquidus line with half-melting injection molding of this invention to generally injecting in metal mold at the molten metal temperature of 30-50 degrees C on melting temperature, injection temperature at least will fall by 30-60 degrees C or more. Therefore, printing to metal mold can be prevented.
[0015] First of all, since it is the coagulation from half-melting and coagulation stress becomes small, it is thought by using this approach that generating of hot tearing can be controlled.
[0016] Especially, the effect affect floating length in 30% or less of rates of solid phase in the half-melting fabricating method becomes remarkable (refer to drawing 8), and such prevention and effectiveness have it in generating of hot tearing. [effective for control] Therefore, in case this half-melting shaping is performed, it is desirable that the rate of solid phase at the time of a half-melting condition is 30% or less. It is thought that seizure and coagulation stress is also advantageous so that the rate of solid phase is generally high, but by this invention approach, since a fluidity will fall if the rate of solid phase is high, lowering of restoration nature and generating of a cold shut tend to take place, and it becomes difficult to obtain a healthy shaping member.
[0017] When especially the mean particle diameter of such solidification structure was 30 micrometers or less, it was found out that especially an elongation value improves greatly.
[0018] Two or less % of the weight and/or 4 or less % of the weight (for example, an yttrium, neodium, a lanthanum, a cerium, a misch metal) of **** elements may be contained for at least one sort of elements chosen from the group which the above-mentioned Magnesium alloy becomes from zinc, manganese, a zirconium, and silicon further. These raise effectively the reinforcement or high temperature strength of the above-mentioned Magnesium alloy below in the upper limit.
[0019]
[Embodiment of the Invention] The whole making machine 1 configuration used for the half-melting fabricating method which starts this invention at drawing 1 is shown. By the shaping approach of this invention, the Magnesium alloy metal grain produced by approaches, such as a cut of a machine, by the hopper 8 in drawing or the raw material 3 of a pellet (3mm or more of diameters) is thrown in. A raw material 3 is supplied in a cylinder 4 through the pass gate 7 of an argon ambient atmosphere from a hopper 8. Within this cylinder 4, while a raw material 3 is ahead sent on a screw 2, it is heated. 10 shows this heating zone. The Magnesium alloy raw material 3 will be in the half-melting condition that solid phase and the liquid phase were intermingled as illustrated, at the temperature below the liquidus line, although whenever [stoving temperature] will be in a melting condition in the abbreviation liquidus line. Moreover, as for the Magnesium alloy in a half-melting condition, the shearing force divides solid phase finely like a graphic display by revolution churning of a screw. Here, when a screw 2 is ahead extruded by the back high-speed injection device 5, in the state of half-melting, finely, high-speed injection will be carried out like [nozzle / 9] a graphic display, and it will fill up with the molten metal to which beating of the solid phase was carried out in metal mold 6. Here, application-of-pressure maintenance of the inside of metal mold is carried out to coagulation, and an aperture shaping product is taken out for the mold after coagulation.
[0020] The iron crucible was installed in one to examples 1-7 and example of comparison 5 low frequency furnace, and the alloy of the component of an example and the example of a comparison was ingoted, making SF6 gas 1% (** being a dried air) flow on a molten metal front face. These alloys were cast on the plate, the pellet of the diameter of 3-5mm was manufactured with milling, and half-melting shaping was performed using the above-mentioned making machine by making these into a raw material.
[0021]

[A table 1]

		化学組成 (重量%)					
		Al	Ca	Si	Mn	Sr	Mg
実施例1	Mg-3Al-2Ca	2.98	2.05	0.30	0.25	−	残部
実施例2	Mg-4Al-2Ca	3.95	2.02	0.30	0.32	−	↑
実施例3	Mg-4Al-3Ca	4.02	3.06	0.25	0.28	−	↑
実施例4	Mg-6Al-3Ca	5.97	3.10	0.28	0.30	−	↑
実施例5	Mg-4Al-2Ca-0.03Sr	3.87	2.06	0.25	0.25	0.03	↑
実施例6	Mg-4Al-2Ca-0.09Sr	4.02	1.98	0.30	0.23	0.09	↑
実施例7	Mg-4Al-2Ca-0.15Sr	4.05	2.10	0.23	0.25	0.15	↑
比較例1	ASTM AS41相当	4.39	−	0.45	0.28	−	↑
比較例2	Mg-9Al-0.5Ca	8.70	0.49	0.90	0.21	−	↑
比較例3	ASTM AZ91D相当	8.84	−	0.02	0.22	−	↑
比較例4	Mg-4Al-4Ca	4.02	3.96	0.32	0.32	−	↑
比較例5	Mg-3Al-3Ca	2.75	2.71	0.27	0.36	−	↑

[0022] Using the machine of 450t of mold clamp force, an injection speed is about 700kg/cm2 in 50 m/s and injection pressure in the metal mold gate section, and, as for both half-melting shaping, the condition set the temperature of the alloy of the nozzle section as the temperature of 550-580 degrees C below the liquidus line. In the above process condition, a test piece for tensile test (JIS No. 4 test piece) is created, and it is JIS. Z 150 degrees C and the creep property in 50MPa were examined with the creep-under-tensile-force test method based on 2271. A result is shown in drawing 2 . It turns out that AS41 presupposed that the Magnesium alloy concerning this invention excels AZ91D of the example 3 of a comparison in creep resistance is excelled in a creep-proof property.
[0023] Moreover, breaking strength and elongation after fracture were measured with the Instron tension tester at a part for 10mm/in crosshead rate, and the measurement temperature of 25 degrees C. A result is shown in a table 2. Although the example 2 of a comparison, aluminum, and calcium with which aluminum exceeds two to range 6 weight of this invention are in the range of this invention, to the example 4 of a comparison for which a calcium/aluminum ratio exceeds 0.8, 2 - 6 % of the weight of aluminum and 0.5 - 4 % of the weight of calcium are contained, and it turns out that a calcium/aluminum ratio shows the elongation excellent in 0.8 or less example.
[0024]
[A table 2]

	Al量 (重量%)	Ca量 (重量%)	伸び (%)
実施例2	3.95	2.02	6.7
実施例3	4.02	3.06	7.0
実施例4	5.97	3.10	5.2
比較例2	8.70	0.49	0.8
比較例4	4.02	3.96	1.2

[0025] Then, in the example and the example of a comparison, the relation between a calcium/aluminum ratio and the above-mentioned elongation was illustrated to drawing 3 . When a calcium/aluminum ratio will exceed 0.8 from now on, it turns out that elongation falls rapidly. Then, when the relation between a calcium/aluminum ratio and the minimum creep strain rate is found, it turns out that it becomes a smaller creep strain rate when a calcium/aluminum ratio is 0.6 or less (example 2), as shown in drawing 6 , and it excels in a creep-proof property further.
[0026] Moreover, when the fluidity of a graphic display was secured and half-melting shaping was performed using the test mold shown in drawing 4 , the result shown in a table 3 was obtained. Although the casting crack arose in the body upper bed overflow side when the calcium/aluminum ratio approached 1 from this, calcium/aluminum did not generate such any casting crack or less in 0.8.
[0027]

[A table 3]

	Ｃａ／Ａｌ重量比	鋳造割れの有無
実施例１	０.６９	無し
実施例２	０.５１	無し
実施例３	０.７６	無し
実施例４	０.５２	無し
比較例１	０.９９	有り
比較例５	０.９９	有り

[0028] if the residence time at the time of casting generally becomes long -- the diameter of solid phase -- rapid -- increasing (example 2 of drawing 5) -- when strontium is added, it turns out that crystal detailed-ized effectiveness can work and buildup of the diameter of solid phase by the residence time can be controlled.
[0029] the fluidity nature assessment shown in drawing 7 using the alloy raw material of an example 2 -- public funds -- change half-melting molding temperature to a mold, changed the rate of solid phase in a molten metal, the molten metal was made to invade towards a graphic display, and the fluidity nature was evaluated. A result is shown in drawing 8 . If 30% of rates of solid phase is exceeded from this result, floating length will descend rapidly. Since this fluidity affects the diameter of organization crystal grain of a shaping member, it turns out that it is desirable to fabricate in the half-melting fabricating method in the condition of 30% or less of rates of solid phase.
[0030] Although it is used in half-melting shaping by making a Magnesium alloy raw material into the gestalt of a metal grain or a pellet Since this metal grain generates the nucleus of a recrystallization grain and increases the diameter of solid phase after carrying out it for a while after heating if processing distortion is given to the interior by cutting etc. If the metal grain which has the case where a metal grain without processing distortion is used, and processing distortion is compared, as shown in drawing 10 , the growth rates of solid phase differ, and he can understand that the way of the latter is excellent in detailed-ization of the diameter of crystal grain of a shaping member.
[0031]
[Effect of the Invention] Since the shaping member which controls a calcium/aluminum ratio in a Mg-aluminum-calcium system heatproof Magnesium alloy member, and is excellent in a hot creep-proof property can be obtained by the above explanation according to this invention so that clearly, engine components, such as transmission components for automobiles, such as a clutch piston and a clutch drum, and a rocker arm, can be manufactured with a lightweight Magnesium alloy, and sufficient endurance can be given. Moreover, in this invention, elongation can be held in equivalent to a conventional method or the ordinary temperature beyond it, and a high-temperature-strength list, solving the technical problem of the seizure to a hot-tearing metallurgy mold by the conventional pressure die casting by carrying out half-melting shaping at the temperature below the liquidus line.

[Translation done.]

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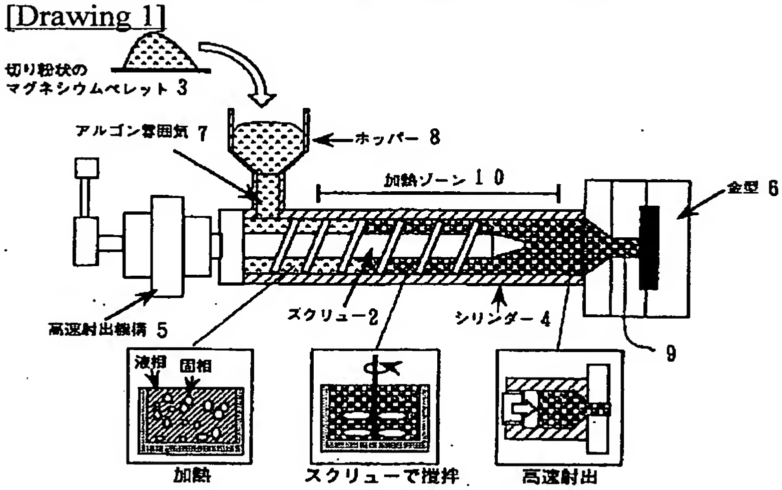
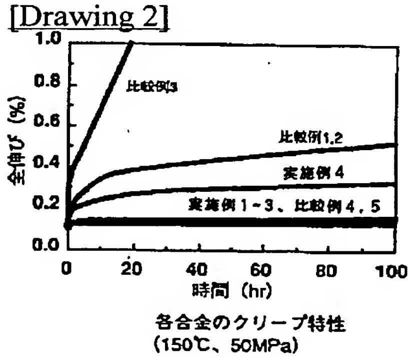
1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

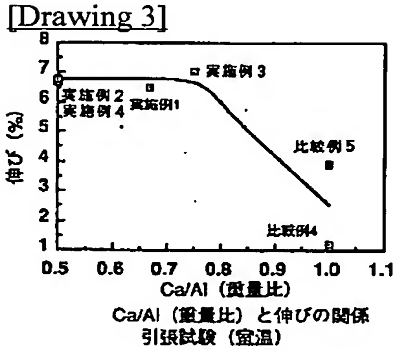
[Drawing 1]



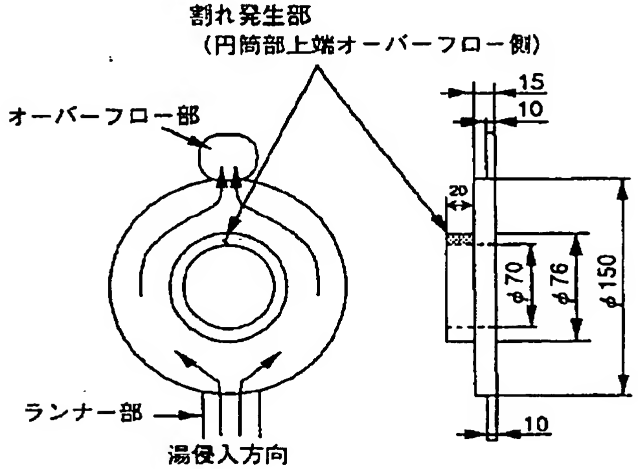
[Drawing 2]



[Drawing 3]

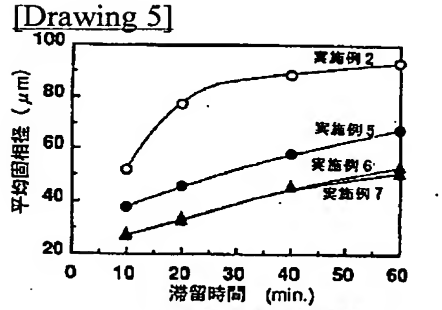


[Drawing 4]



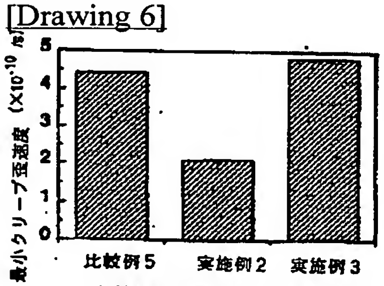
鋳造割れ試験用金型

[Drawing 5]



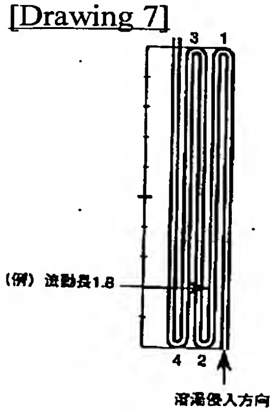
固相径と滞留時間の関係

[Drawing 6]



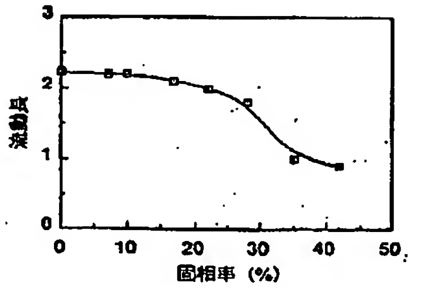
各種合金の最小クリープ歪速度

[Drawing 7]



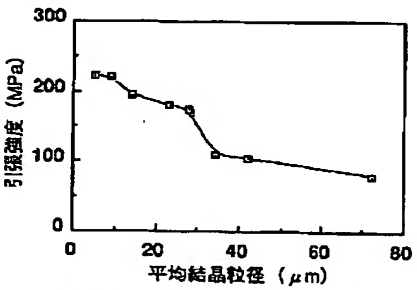
湯流れ性評価用金型

[Drawing 8]



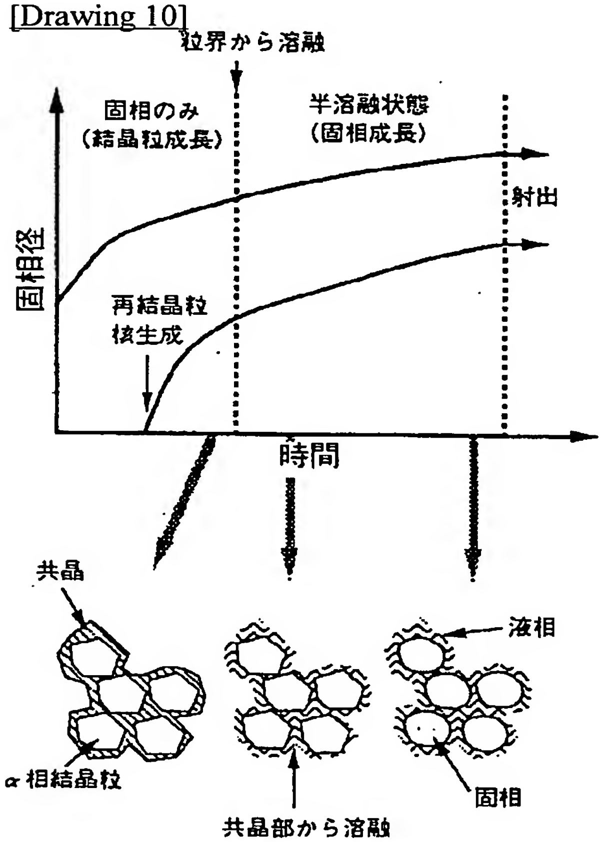
実施例２成形時の流動長と固相率の関係

[Drawing 9]



実施例３の引張強度と平均結晶粒径の関係

[Drawing 10]



固相の成長模式図

[Translation done.]

Original document

HEAT RESISTANT MAGNESIUM ALLOY MOLDED MEMBER, HEAT RESISTANT MAGNESIUM ALLOY USED FOR THE MOLDING AND MOLDING METHOD THEREFOR

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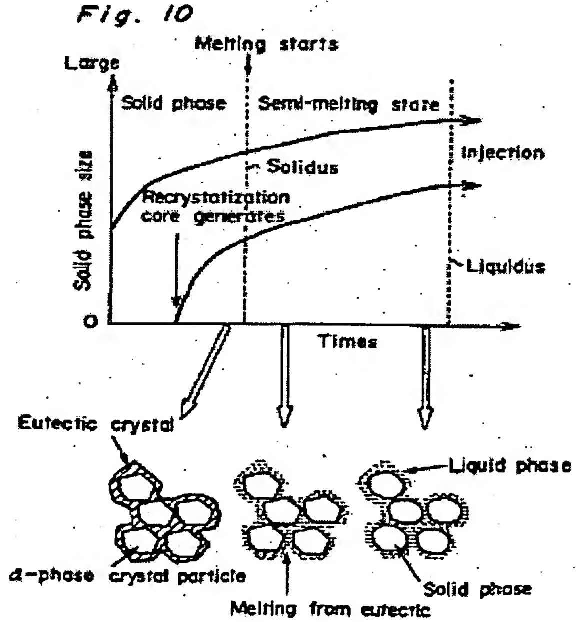
View INPADOC patent family

Abstract of **JP9272945**

PROBLEM TO BE SOLVED: To produce a heat resistant Mg allay molded member excellent in moldability and extensibility while its creep resistance is secured, in an Mg-Al- Ca-Mn alloy, by specifying its compsn. and regulating the ratio of Ca/Al to a specified value or below. SOLUTION: An Mg alloy having a compsn. contg., by weight, 2 to 6% Al, 0.5 to 4% Ca, and the balance Mg, and in which the ratio of Ca/Al is regulated to <=0.8 is subjected to half-melting injection molding at a liquids temp. or below in which solid phases and liq. phases are coexistent. In this way, the heat resistant Mg alloy molded member such as automobile transmission parts or engine parts excellent in creep resistance can be obtd. Al is required by >=2%, but, in the case of >6%, its elongation deteriorates even if the half- melting injection molding is executed. Ca is added for increasing its high temp. strength tending toward reduction accompanying the addition of Mg to Al, but, for preventing the reduction of its moldability and the elongation of the molded member, the ratio of Ca/Al is required to be suppressed to <=0.8. Moreover, <=0.15% Sr is preferably added as a refining agent.

Fig. 10



Description of corresponding document: **EP0799901**

The present invention relates to a heat-resistant magnesium alloy member having an excellent molding property and an excellent elongation property while keeping creep resistance property, its starting alloy compound, and a method of preparing the heat-resistant magnesium alloy member.

Magnesium alloy is the most low density one of the metal materials which are in practically use at present, and is strongly expected as a lightweight material for automobiles in future. The magnesium alloy which is most popularly used at present is Mg-Al-Zn-Mn alloy (e.g., AZ91D alloy), and as it has a high strength at a room temperature and a good corrosion resistance, it is applied to transmission cases for an automobile, cylinder head covers, and the like. However, it has such defects that, at a temperature range exceeding 120 DEG C, it begins to show loss of strength characteristics, and especially becomes inferior in creep resistance, leading to a problem of yielding of bearing surface of the screw tightening part on the level of the packaged product.

On the other hand, as an aluminum alloy having an improved heat-resistance, there is used Mg-Al-Si AS41 magnesium alloy. However, though said alloy shows better creep resistance than the above AZ91D, it shows insufficient characteristics in the neighborhood of 150 DEG C of the use temperature, and moreover, as it shows low tensile strength characteristics at both room temperature and high temperature, it is required to be of thick wall to secure the required strength, thereby providing a problem of lowering the weight lightening effect due to magnesium materials.

Besides, there are alloys such as QE22 with addition of silver or rare earth metals to improve a heat resistance thereof, but they have defects of being expensive and not suited to die-cast due to a poor casting property.

For the above reasons, there came to be newly proposed Mg-Al-Ca-Mn alloy (Japanese Laid-open Patent Publication HEI6-25790/1994) having excellent strength at high temperature. Here, it is said that, especially when the Ca/Al ratio is set to be more than 0.7, preferably more than 0.75, precipitates to be crystallized in the magnesium alloy convert into Mg-Ca compounds which crystallize, resulting in production of high temperature strength characteristics.

However, it has been found that, in a case of die-casting a member with a magnesium alloy having a high Ca/Al ratio, there often occur hot cracks, and due to a high melting temperature there easily occurs seizure to the metal mold.

In view of the problems held by the conventional techniques as above, a first object of the present invention is to provide a heat-resistant magnesium alloy member having excellent molding property and elongation while maintaining the physical properties, especially creep resistance, suited to the engine parts of automobiles and the like.

A second object of the present invention is to provide a pertinent molding method for preparing the above heat-resistant magnesium alloy member in place of conventional die-cast methods.

Further, a third object of the present invention is to provide an alloy composition suited for producing a heat-resistant magnesium alloy member having the excellent molding property and elongation while maintaining the creep resistance.

As a result of the repeated reviews to solve the above problems, the present inventors have found out that, in the Al-Ca magnesium alloy, when a semi-solid molding method of injection molding under the state of solid phase and liquid phase being present in mixture is applied in place of the die-cast method, the seizure of metal mold can be prevented, and also an excellent strength can be imparted to the molded member. However, in order to maintain the state of presence in mixture of solid phase and liquid phase, it is necessary to increase the addition amount of aluminum as large as possible.

On the other hand, aluminum dissolves in magnesium in solid state and shows age-hardening, and it is added to increase the mechanical properties of alloy, but it is recommended to add calcium so as to maintain the Ca/Al ratio to 0.7 or more to strengthen the high temperature strength which is in a tendency to be lowered

by the addition of Al to Mg (Japanese Patent Laid-open Publication HEI6-25790/1994). However, when the Ca amount is large, casting cracks and seizure to metal mold often occur in molding, and additionally, a large amount of Mg-Ca compound crystallizes, with the result that there is a tendency of lowering of the elongation of the molded product, and it has been found that it is rather necessary to make the Ca/Al ratio no more than 0.8.

Accordingly, in the present invention, based on the above two findings, there is provided a magnesium alloy molding member comprising 2 to 6% by weight of aluminum and 0.5 to 4% by weight of calcium, and the balance of magnesium and unavoidable impurities, wherein a Ca/Al ratio is no more than 0.8, to have an excellent anti-creep property, molding property, and elongation.

In general, in the magnesium alloy, in order to obtain solid phase dissolution in magnesium, to exhibit age-hardening, and to elevate mechanical strength, it has been understood to be preferable to add 2 - 10% by weight of aluminum. While it is necessary in the present invention to add more than 2% by weight of aluminum, when the amount of addition exceeds 6% by weight, it has been found that the elongation is lowered even if the semi-solid injection molding would be carried out. Accordingly, in order to obtain the designed effect while carrying out the semi-solid injection molding, it has been found that the addition amount should be limited to no more than 6% by weight.On the other hand, calcium is added to increase the high temperature strength which is in a tendency to be lowered by the addition of aluminum to magnesium, but it has been found that it is necessary to suppress the Ca/Al ratio to no more than 0.8 to prevent lowering of the molding property and elongation of the molding member, and in addition, the Ca amount should be limited to 0.5 - 4% by weight.

Strontium is used as a micronizing agent in the casting of magnesium, and as it can display the micronizing effect in solid phase in the semi-solid injection molding of the present invention, it is preferably added. The suitable addition amount is no more than 0.15% by weight.

The above molding member shows the crystal particle size of no more than 30 mu m with the tensile strength of 180 Mpa (298 DEG K; ref. Fig. 9) or more, and excellent creep resistance of the minimum creep rate of no more than 4 x 10<-10>/S under the test temperature of 150 DEG C and the test load of 50 MPa (according to JIS Z 2271 "method of tensile creep test of metal material"). Accordingly, it is suitable for the transmission part or engine part for automobiles. Especially, when the Ca/Al ratio is no more than 0.6, the molding member shows a more excellent creep resistance.

The present invention is to provide a heat-resistant magnesium alloy material to be molded by a semi-solid injection molding while maintaining excellent creep resistance property with the excellent molding property and elongation, comprising as an alloy material to be used for molding the above magnesium alloy molding part heat-resistant magnesium comprising 2 to 6% by weight of aluminum and 0.5 to 4% by weight of calcium, and the balance of magnesium and unavoidable impurities, and preferably further Sr of no more than 0.15% by weight, with adjustment, if necessary, of a Ca/Al ratio of no more than 0.8, preferably a Ca/Al ratio of no more than 0.6.

Especially, as for the alloy material, in case of molding by a semi-solid injection molding method, it has been found that the material in the form of metal particles or pellets into which internal strain is introduced is effective for micronizing the crystals (ref. Fig. 10). As for the processing method for the metal particles or pellets, a cutting method is advantageous costwise.

Further, in case of applying a semi-solid injection molding wherein an injection molding is carried out in the state of a solid phase and a liquid phase being present in mixture, practice can be made at a temperature lower than a liquidus temperature. Accordingly, the present invention is to provide a method for molding a heat-resistant magnesium alloy member characterized by carrying out a semi-solid injection molding, while maintaining an excellent creep resistance property with having an excellent molding property and elongation.

Against the fact that the die-cast method is in general to make injection into the metal mold at a temperature of 30 - 50 DEG C above a melting temperature, in the semi-solid injection molding of the present invention, injection can be made at a temperature higher than the solidus temperature of the alloy and lower than the liquidus temperature, and accordingly the injection temperature is lowered by at least 30 - 60 DEG C, so that

the seizure to the metal mold can be prevented.

It can be understood that, since solidification takes place from a semi-solid state in the present invention, and coagulation stress therewith becomes small, generation of hot cracking can be prevented due to this method mechanism.

Especially, in the semi-solid molding method, in the range of no more than 30% by weight of the solid phase rate, these prevention and effect on a flow length become remarkable (ref. Fig. 8), and the generation of hot cracking can be effectively prevented. Accordingly, in case of carrying out the semi-solid molding, the solid phase ratio in the semi-solid state is preferably no more than 30%. In general, it has been understood that a higher solid phase ratio is more advantageous for the seizure and coagulation stress, but in the present invention method, when the solid phase rate is high, the fluidity is lowered to give a tendency of lowering in filling property and generation of cold shut, thereby making it difficult to obtain a sound molding member.

It has been found that, especially when the average particle size of these coagulation textures is no more than 30 mu m, the elongation amount shows specially large improvement.

The above magnesium alloy may further contain no more than 2% by weight of at least one element selected from the group consisting of zinc, manganese, zirconium, and silicon, and/or no more than 4% by weight of a rare earth metal (e.g., yttrium, neodymium, lanthanum, cerium, misch metal). These are to improve the strength or high temperature strength of the above magnesium alloy effectively in the range no more than the upper limit thereof.

Fig. 1 is a schematic diagram showing the constitution of the molding machine to be used for the semi-solid molding process and injection molding process according to the present invention.

Fig. 2 is a graph for making comparison of the creep characteristics of various magnesium alloy molding members.

Fig. 3 is a graph to show the relations between the Ca/Al ratio and the elongation at room temperature in various magnesium alloy molding members.

Fig. 4 is a schematic diagram showing a metal mold for testing casting cracks.

Fig. 5 is a graph showing the relation between the solid phase diameter and the staying time.

Fig. 6 is a graph showing the minimum creep strain rates of various magnesium alloy molding members.

Fig. 7 is a schematic diagram showing the metal mold for evaluating the flowing properties of various magnesium alloys.

Fig. 8 is a graph showing the relations between the solid phase ratio and the flowing length in the alloy composition in Example 2 measured by using a metal mold of Fig. 7.

Fig. 9 is a graph showing the relations between the average crystal particle size and the tensile strength of the member molded from the alloy composition of Example 3.

Fig. 10 is a schematic diagram showing the solid phase growth stages in the cases of using the metal particles having no work strain and those having the work strain.

In Fig. 1, there is shown the whole constitution of the molding machine 1 to be used for the semi-solid molding method according to the present invention. In the molding method of the present invention, the material 3 of magnesium alloy metal particles or pellets (more than 3 mm in diameter) manufactured by the method of cutting or the like is charged into the hopper 8 in the drawing. The material 3 is supplied to the cylinder 4 from the hopper 8 through the inlet 7 of argon atmosphere. In this cylinder 4, the material 3 is heated while being sent forward by the screw 2. This heating zone is shown by the mark 10. At an approximate liquidus temperature of heating, the magnesium alloy material 3 shows a molten state, but at a level lower than the liquidus temperature the material becomes semi-solid condition in which the solid phase

and the liquid phase are present in mixture, as illustrated. Also, in the magnesium alloy which is in a semi-solid condition, its shearing force acts to separate the solid phase finely as illustrated by agitation by the screw rotation. Here, when the screw 2 is pushed forward with the rear high speed injection mechanism 5, the molten material in which the solid phase has been finely cut under the semi-solid state is injected at high speed from the nozzle 9 as illustrated and filled in the metal mold 6.Here, the contents in the metal mold are held under pressure until solidification, and thereafter the metal mold is opened to take out the molding product.

Examples 1 - 7 and Comparative Examples 1 - 5

An iron crucible is installed in a low frequency furnace, and while flowing 1% of the SF6 gas (rest is dry air) on the surface of the molten material, the alloys having the components of Examples and Comparative Examples were prepared by melting. The resulting alloys were cast on a plate to prepare 3 - 5 mm diameter pellets by milling.Using these as raw materials, semi-solid molding was carried out by using the above molding machine.
<tb><TABLE> Id=[Table 1] Columns=8 OR=L
<tb>
<tb>Head Col 1:
<tb>Head Col 2:
<tb>Head Col 3 to 8: Chemical Composition (Wt.%)
<tb>
<tb>SubHead Col 1:
<tb>SubHead Col 2:
<tb>SubHead Col 3: Al
<tb>SubHead Col 4: Ca
<tb>SubHead Col 5: Si
<tb>SubHead Col 6: Mn
<tb>SubHead Col 7: Sr
<tb>SubHead Col 8:Mg
<tb>Example 1<SEP>Mg-3Al-2Ca<SEP>2.98<SEP>2.05<SEP>0.30<SEP>0.25<SEP>-<SEP>Remainder
<tb>Example 2<SEP>Mg-4Al-2Ca<SEP>3.95<SEP>2.02<SEP>0.30<SEP>0.32<SEP>-<SEP> &uarr&
<tb>Example 3<SEP>Mg-4Al-3Ca<SEP>4.02<SEP>3.06<SEP>0.25<SEP>0.28<SEP>-<SEP> &uarr&
<tb>Example 4<SEP>Mg-6Al-3Ca<SEP>5.97<SEP>3.10<SEP>0.28<SEP>0.30<SEP>-<SEP> &uarr&
<tb>Example 5<SEP>Mg-4Al-2Ca-0.03Sr<SEP>3.87<SEP>2.06<SEP>0.25<SEP>0.25<SEP>0.03<SEP> &uarr&
<tb>Example 6<SEP>Mg-4Al-2Ca-0.09Sr<SEP>4.02<SEP>1.98<SEP>0.30<SEP>0.23<SEP>0.09<SEP> &uarr&
<tb>Example 7<SEP>Mg-4Al-2Ca-0.15Sr<SEP>4.05<SEP>2.10<SEP>0.23<SEP>0.25<SEP>0.15<SEP> &uarr&
<tb>Comparative Example 1<SEP>ASTM AS41 Equivalent<SEP>4.39<SEP>-<SEP>0.45<SEP>0.28<SEP>-<SEP> &uarr&
<tb>Comparative Example 2<SEP>Mg-9Al-0.5Ca<SEP>8.70<SEP>0.49<SEP>0.90<SEP>0.21<SEP>-<SEP> &uarr&
<tb>Comparative Example 3<SEP>ASTM AZ91D Equivalent<SEP>8.84<SEP>-<SEP>0.02<SEP>0.22<SEP>-<SEP> &uarr&
<tb>Comparative Example 4<SEP>Mg-4Al-4Ca<SEP>4.02<SEP>3.96<SEP>0.32<SEP>0.32<SEP>-<SEP> &uarr&
<tb>Comparative Example 5<SEP>Mg-3Al-3Ca<SEP>2.75<SEP>2.71<SEP>0.27<SEP>0.36<SEP>-<SEP> &uarr&
<tb></TABLE>

For the semi-solid molding, a machine having the clamping force of 450 t was used under the conditions of injection speed at the metal mold gate part of 50 m/s, injection pressure of about 700 kg/cm<2>, and the temperature of the alloy at the nozzle part was set to be lower than the liquidus level of 550 - 580 DEG C. Under the above molding conditions a tensile test piece (JIS No.4 test piece) was prepared, with which the creep property at 150 DEG C, 50 MPa was examined by the tensile creep test method based on JIS Z 2271.

The results are shown in Fig. 2. It can be seen that the magnesium alloy of the present invention is more excellent in creep resistance characteristic than AS41 which is commented as being superior in creep resistance to AZ91D of Comparative Example 3.

Further, the breaking strength and breaking elongation were measured with an instron tensile tester at a cross head rate of 10 mm/min. and at a measuring temperature of 25 DEG C. The results are shown in Table 2. It can be seen that, in comparison with Comparative Example 2 in which the aluminum content exceeds the present invention range of 2 - 6% by weight and Comparative Example 4 in which the aluminum and calcium contents lie within the present invention range but the Ca/Al ratio exceeds by 0.8, the Examples containing 2 - 6% by weight of aluminum and 0.5 - 4% by weight of calcium and having the Ca/Al ratio of no more than 0.8 show excellent elongation.
<tb><TABLE> Id=[Table 2] Columns=4
<tb>
<tb>Head Col 1:
<tb>Head Col 2: Al amount (Wt.%)
<tb>Head Col 3: Ca amount (Wt.%)
<tb>Head Col 4:Elongation (%)
<tb>Example 2<SEP>3.95<SEP>2.02<SEP>6.7
<tb>Example 3<SEP>4.02<SEP>3.06<SEP>7.0
<tb>Example 4<SEP>5.97<SEP>3.10<SEP>5.2
<tb>Comparative Example 2<SEP>8.70<SEP>0.49<SEP>0.8
<tb>Comparative Example 4<SEP>4.02<SEP>3.96<SEP>1.2
<tb></TABLE>

Then, in Examples and Comparative Examples, the relations between the Ca/Al ratio and the above elongation are illustrated in Fig. 3, from which it can be seen that when the Ca/Al ratio exceeds 0.8, the elongation is sharply lowered. In this connection, when the relations between the Ca/Al ratio and the minimum creep rate of strain are observed, as shown in Fig. 6, in case of the Ca/Al ratio being no more than 0.6 (Example 2), the smaller creep rate of strain is shown, and it can be seen that the creep resistance property becomes more excellent.

Further, when the semi-melting molding was carried out by using the metal mold for test as shown in Fig. 4 and securing the illustrated running, there were obtained the results as shown in Table 3. As a result, it was seen that when the Ca/Al ratio approached 1, casting cracks were formed on the overflow side at the top end of the cylindrical part, but at the Ca/Al ratio of no more than 0.8, no such casting crack was formed at all.
<tb><TABLE> Id=[Table 3] Columns=3
<tb>
<tb>Head Col 1:
<tb>Head Col 2: Ca/Al weight ratio
<tb>Head Col 3: Casting crack formed or not
<tb>Example 1<SEP>0.69<SEP>No
<tb>Example 2<SEP>0.51<SEP>No
<tb>Example 3<SEP>0.76<SEP>No
<tb>Example 4<SEP>0.52<SEP>No
<tb>Comparative Example 1<SEP>0.99<SEP>Yes
<tb>Comparative Example 5<SEP>0.99<SEP>Yes
<tb></TABLE>

In general, when the staying time in casting is extended, the solid phase diameter is sharply increased (Example in Fig. 5), but it can be seen that, when strontium is added, the crystal micronizing effect is actuated to suppress the increment in the solid phase diameter attributed to the staying time.

Using the alloy material of Example 2, the semi-solid molding temperature was varied in the metal mold for evaluating flowing property as shown in Fig. 7, the molten material was introduced in the illustrated direction, and its flowing property was evaluated. The results are shown in Fig. 8. From the results it can be seen that, when the solid phase rate exceeds 30%, the flow length is sharply lowered, and as this flow gives effect on the particle size of the texture crystals of the molding material, desirably the molding is made under the solid phase condition of no more than 30% in the semi-solid molding method.

In the semi-solid molding, the magnesium alloy material is used in the form of the metal particles or pellets. When work strain is given inside the metal particles by cutting work or the like, the metal particles form the nuclei of recrystallization shortly after the heating, and increase the solid phase diameter. Therefore, when comparison is made between the case of using the metal particles having no work strain and that of using the metal particles having work strain, it can be understood that the growth rates of the solid phase are different as shown in Fig. 10, and the latter is superior to the former in the point of micronization of the crystal particle size of the molding member.

As will be apparent from the above description, according to the present invention, it is possible to obtain a molding member having excellent creep resistance characteristic at high temperature by controlling Ca/Al ratio in Mg-Al-Ca heat resistant magnesium alloy member. Therefore, it is possible to produce the transmission parts for automobiles such as clutch piston and clutch drum and engine parts such as rocker arm with the lightweight magnesium alloy to give a sufficient durability.

Further, according to the present invention, by carrying out semi-solid molding at a temperature lower than the liquidus level, the problems of hot crack and seizure to the metal mold which had been remarkable in the conventional die-cast process are dissolved, and on the other hand, the strength at room temperature and high temperature along with elongation equivalent to or higher than those of the conventional process can be retained.

Claims of corresponding document: **EP0799901**

1. A heat-resistant magnesium alloy member having excellent creep resistance property which comprises 2 to 6% by weight of aluminum and 0.5 to 4% by weight of calcium, and the balance of magnesium and inevitable impurities, wherein a Ca/Al ratio thereof is no more than 0.8.

2. The heat-resistant magnesium alloy member according to Claim 1, having a Ca/Al ratio of no more than 0.6, and creep resistance property of no more than 4 x 10<-10>/S of minimum creep rate under the test temperature of 150 DEG C and the test load of 50 Mpa.

3. The heat-resistant magnesium alloy member according to Claim 1 or 2, wherein the magnesium alloy further contains no more than 0.15% by weight of Sr.

4. The heat-resistant magnesium alloy member according to any one of Claims 1 to 3, wherein the average particle size of the crystals is no more than 30 mu m.

5.The heat-resistant magnesium alloy member according to any one of Claims 1 to 4, wherein the molding parts are the transmission parts or engine parts for automobiles.

6. A heat-resistant magnesium alloy composition comprising 2 to 6% by weight of aluminum and 0.5 to 4% by weight of calcium, and the balance of magnesium and inevitable impurities, which gives excellent creep resistance property by a semi-solid injection molding at a temperature range between a solidus temperature of the alloy and a liquidus temperature of the alloy wherein the solid phase and the liquid phase are present in mixture.

7. The heat-resistant magnesium alloy composition according to Claim 6, wherein the magnesium alloy further contains no more than 0.15% by weight of Sr.

8. The heat-resistant magnesium alloy composition according to Claim 6 or 7, having a Ca/Al ratio of no more than 0.8.

9.The heat-resistant magnesium alloy composition according to Claim 8, having a Ca/Al ratio of no more than 0.6.

10. The heat-resistant magnesium alloy composition according to any one of Claims 6 to 9, being in the form of metal particles or pellets into which internal strain is introduced.

11. A method of molding a heat-resistant magnesium alloy having excellent creep resistance property, which comprises preparing an alloy composition comprising 2 to 6% by weight of aluminum and 0.5 to 4% by weight of calcium, and the balance of magnesium and inevitable impurities;
subjecting said alloy composition to a semi-solid injection molding at a temperature range between a solidus temperature of the alloy and a liquidus temperature of the alloy wherein a solid phase and a liquid phase are present in mixture.

12. The method of molding a heat-resistant magnesium alloy member according to claim 11, wherein the solid phase rate in semi-melt state is no more than 30% at the time of carrying out an injection molding.

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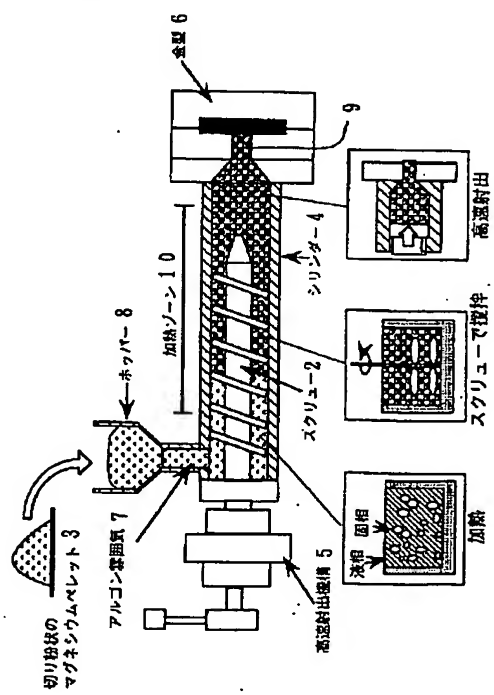
最終頁に続く

(54)【発明の名称】　耐熱マグネシウム合金成形部材、その成形に用いる耐熱マグネシウム合金および該成形方法

(57)【要約】

【課題】　耐クリープ特性を確保しつつ、特に成形性、伸び率に優れる耐熱マグネシウム合金成形部材、その成形方法およびそれに用いる合金組成を提供すること。

【解決手段】　アルミニウム２～６重量％及びカルシウム０．５～４重量％を含有し、残部がマグネシウムと不可避の不純物からなり、Ｃａ／Ａｌ比が０．８、好ましくは０．６以下のマグネシウム合金を液相線温度以下で半溶融射出成形する。



【特許請求の範囲】
【請求項１】　アルミニウム２～６重量％及びカルシウム０．５～４重量％を含有し、残部がマグネシウムと不可避の不純物からなり、Ｃａ／Ａｌ比が０．８以下のマグネシウム合金から成形された成形部材であり、耐クリープ性に優れる耐熱マグネシウム合金成形部材。
【請求項２】　Ｃａ／Ａｌ比が０．６以下で、試験温度１５０℃、試験荷重５０ＭＰａでの最小クリープ速度４×10^{-10}／Ｓ以下の耐クリープ性を有する請求項１記載の耐熱マグネシウム合金成形部材。
【請求項３】　マグネシウム合金が更に０．１５重量％以下のＳｒを含有する請求項１または２に記載の耐熱マグネシウム合金成形部材。
【請求項４】　平均結晶粒径が３０μm以下である請求項１ないし３のいずれかに記載の耐熱マグネシウム合金成形部材。
【請求項５】　成形部品が自動車用トランスミッション部品またはエンジン部品である請求項１ないし４のいずれかに記載の耐熱マグネシウム合金成形部材。
【請求項６】　アルミニウム２～６重量％及びカルシウム０．５～４重量％を含有し、残部がマグネシウムと不可避の不純物からなり、固相と液相が混在する液相線以下の温度での半溶融射出成形によって優れた耐クリープ性が得られる耐熱マグネシウム合金。
【請求項７】　マグネシウム合金が更に０．１５重量％以下のＳｒを含有する請求項６に記載の耐熱マグネシウム合金。
【請求項８】　Ｃａ／Ａｌ比が０．８以下である請求項６または７に記載の耐熱マグネシウム合金。
【請求項９】　Ｃａ／Ａｌ比が０．６以下である請求項８に記載の耐熱マグネシウム合金。
【請求項１０】　内部歪みを導入した金属粒またはペレット形態である請求項６ないし９のいずれかに記載の耐熱マグネシウム合金。
【請求項１１】　請求項６ないし１０のいずれかに記載のマグネシウム合金を固相と液相の混在する液相線温度以下で半溶融射出成形することを特徴とする耐クリープ性に優れる耐熱マグネシウム合金成形部材の成形方法。
【請求項１２】　半溶融射出成形を行う際、半溶融状態時の固相率が３０％以下である請求項１１記載の耐熱マグネシウム合金部材の成形方法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は耐クリープ性を確保しつつ、特に成形性、伸び性に優れる耐熱マグネシウム合金成形部材、その成形に用いる耐熱マグネシウム合金およびその成形方法に関するものである。
【０００２】
【従来の技術】マグネシウム合金は現在実用化されている金属材料の中でも最も低密度であり、今後の自動車用軽量材として強く期待されている。現在最も一般的に用いられているマグネシウム合金はＭｇ－Ａｌ－Ｚｎ－Ｍｎ系合金（例えばＡＺ９１Ｄ合金）であり、室温強度が高く、耐食性も良好であるため、自動車用トランスミッションケース、シリンダヘッドカバーなどに適用されている。しかしながら、１２０℃を越える温度域では強度特性が低下し始め、特に耐クリープ性が劣るという欠点があり、実部品レベルではネジ締結部の座面がへたるなどの問題に繋がる。
【０００３】他方、耐熱性を改善した合金としてはＭｇ－Ａｌ－Ｓｉ系のＡＳ４１マグネシウム合金が使用されている。しかしながら、耐クリープ性に関しては上記ＡＺ９１Ｄよりも良好であるが、使用温度が１５０℃近傍では不十分な特性となり、しかも室温、高温とも引張強度特性が低いため、要求強度を確保するには厚肉とせざるを得ず、マグネシウムの軽量化効果が減少するという問題が生ずる。
【０００４】その他、耐熱性を改善した合金として銀や希土類元素などを添加したＱＥ２２などの合金があるが、高価であり、鋳造性の点でダイキャストには適さないなどの欠点がある。
【０００５】そのため、新たに高温強度に優れる、Ｍｇ－Ａｌ－Ｃａ－Ｍｎ系合金（特開平６－２５７９０号）が提案されるに至った。ここでは、特にＣａ／Ａｌの比を０．７、好ましくは０．７５以上にするとマグネシウム合金中に晶出する析出物の組織形態が変化し、Ｍｇ－Ｃａ化合物が晶出して優れた高温強度特性を示すようになるとしている。
【０００６】
【発明が解決しようとする課題】しかしながら、Ｃａ／Ａｌ比の高いマグネシウム合金ではダイキャストにおいて部材を成形しようとする場合、熱間割れが発生しやすく、溶湯温度が高いと金型への焼付きが発生しやすいなどの問題を残している。本発明は、このような従来技術の有する課題に鑑み、自動車などのエンジン部品等に適する物性、特に耐クリープ性を確保しつつ、成形性、伸び性に優れる耐熱マグネシウム合金成形部材を提供することを第１の目的とする。本発明の第２の目的は汎用されるダイキャストに代え、上記耐熱マグネシウム合金成形部材の適切な成形方法を提供することにある。更に、本発明の第３の目的は上記耐クリープ性を確保しつつ、成形性、伸び性に優れる耐熱マグネシウム合金成形部材を製造するに適する合金組成を提供することにある。
【０００７】
【課題を解決するための手段】本発明者らは上記の課題を解決するために種々検討を重ねた結果、Ａｌ－Ｃａ系マグネシウム合金ではダイキャスト法に代え、固相と液相が混在した状態で射出成形を行う半溶融成形法を適用とすると、金型の焼き付きを防止できると同時に、成形される部材に優れた強度を付与することができることを

見い出したが、その固相と液相が混在した状態を維持するためにはアルミニウムの添加量を極力増大することが必要となる。他方、アルミニウムはマグネシウムに固溶し、時効硬化性を示し、合金の機械的性質を高めるために添加するが、マグネシウムへのアルミニウムの添加に伴い、低下する傾向にある高温強度を増強するためにＣａ／Ａｌ比を０．７以上に保持するようにカルシウムを添加することが推奨されている（特開平６－２５７９０号）。しかしながら、このカルシウム量が多いと、成形時に鋳造割れ及び金型への焼き付きが発生し易い上、Ｍｇ－Ｃａ系化合物が多量に晶出するため、成形物の伸びが低下する傾向があり、むしろＣａ／Ａｌ比を０．８以下にする必要があることを見い出した。

【０００８】そこで、本発明は、上記両者の知見に基づき、アルミニウム２～６重量％及びカルシウム０．５～４重量％を含有し、残部がマグネシウムと不可避の不純物からなり、Ｃａ／Ａｌ比が０．８以下のマグネシウム合金から成形された成形部材であり、耐クリープ性を確保しつつ、成形性、伸び性に優れるマグネシウム合金成形部品を提供することにある。一般に、マグネシウム合金ではマグネシウムに固溶し、時効硬化性を示し、合金の機械的性質を高めるためにアルミニウム２～１０重量％を添加するのが好ましいとされている。これに対し、本発明ではアルミニウムは２重量％以上添加される必要があるが、６重量％を越えると、半溶融射出成形を行っても伸びが低下する。そのため、半溶融射出成形を行いつつ所期の効果を達成するため、６重量％以下に制限される。他方、カルシウムはマグネシウムへのアルミニウムの添加に伴い、低下する傾向にある高温強度を増強するために添加されるが、成形性と成形部材の伸びを低下しないようにＣａ／Ａｌ比を０．８以下に押さえる必要があり、しかも０．５～４重量％に制限される。

【０００９】ストロンチウムはマグネシウムの鋳造において微細化剤として使用されるが、本発明に係る半溶融射出成形法においても固相の微細化効果を発揮することができるので、添加するのが好ましい。添加量は０．１５重量％以下が適当である。

【００１０】上記成形部品は結晶粒径が３０μm以下で引張強度１８０ＭＰａ（２９８°Ｋ：図９参照）以上にあり、しかも試験温度１５０℃、試験荷重５０ＭＰａでの最小クリープ速度4×10^{-10}／Ｓ以下である優れた耐クリープ性を示す（ＪＩＳＺ　２２７１「金属材料の引張クリープ試験方法」による）。したがって、自動車用トランスミッション部品またはエンジン部品に適する。特に、Ｃａ／Ａｌ比が０．６以下である場合は耐クリープ性に優れる。

【００１１】また、本発明では、上記マグネシウム合金成形部品の成形に用いる合金素材として、アルミニウム２～６重量％及びカルシウム０．５～４重量％を含有し、残部がマグネシウムと不可避の不純物、好ましくは更に０．１５重量％以下のＳｒを含有してなり、要すれば、Ｃａ／Ａｌ比が０．８以下、好ましくはＣａ／Ａｌ比が０．６以下に調整された、半溶融射出成形によって優れた耐クリープ性を確保しつつ、成形性、伸び性に優れる耐熱マグネシウム合金を提供しようとするものでもある。

【００１２】特に、合金素材としては、半溶融射出成形法で成形する場合、内部歪みを導入した金属粒またはペレット形態であるのが結晶微細化に有効であることが見い出されている（図１０参照）。その加工法としては切削加工がコスト的に有利である。

【００１３】さらに、ダイキャスト法に代え、固相と液相が混在した状態で射出成形を行う半溶融射出成形法を適用とする場合、ダイキャスト法より低温の液相線以下の温度で実施することができる。したがって、本発明は上記記載のマグネシウム合金を液相線温度以下の固相と液相の混在する状態で半溶融成形することを特徴とする耐クリープ性を確保しつつ、成形性、伸び性に優れる耐熱マグネシウム合金部材の成形方法を提供するものでもある。

【００１４】ダイキャストは一般的に溶融温度上３０～５０℃の溶湯温度で金型中に射出するのに対し、本発明の半溶融射出成形では液相線以下の温度で射出するため、少なくとも射出温度は３０～６０℃以上低下することになる。したがって、金型への焼き付きを防止できることになる。

【００１５】そもそも半溶融からの凝固であるので凝固応力が小さくなることからこの方法を使用することにより熱間割れの発生を抑制することができると思われる。

【００１６】特に、これらの防止および効果は半溶融成形法において固相率３０％以下において流動長に及ぼす影響が顕著となり（図８参照）、熱間割れの発生を抑制に効果的である。したがって、この半溶融成形を行う際、半溶融状態時の固相率が３０％以下であるのが好ましい。一般に固相率が高いほど焼付きも凝固応力も有利と思われるが、本発明方法では固相率が高いと流動性が低下するため、充填性の低下や湯境いの発生が起こり易く、健全な成形部材を得ることが困難となる。

【００１７】特にこれらの凝固組織の平均粒径が３０μm以下の時、特に伸び値が大きく向上することが見い出された。

【００１８】上記マグネシウム合金が更に亜鉛、マンガン、ジルコニウム、及びケイ素からなる群から選ばれた少なくとも１種の元素を２重量％以下、及び／又は希土類元素（例えば、イットリウム、ネオジウム、ランタン、セリウム、ミッシュメタル）４重量％以下を含有してもよい。これらはその上限以下で上記マグネシウム合金の強度または高温強度を有効に向上させるものである。

【００１９】

【発明の実施の形態】図１に本発明に係る半溶融成形法に用いられる成形機１の全体構成を示す。本発明の成形方法では、図中のホッパー８に機械の切削等の方法で作製されたマグネシウム合金金属粒またはペレット（径３ｍｍ以上）の原料３を投入する。原料３はホッパー８からアルゴン雰囲気の通入口７を通ってシリンダ４内に供給される。このシリンダ４内ではスクリュー２によって原料３は前方に送られながら、加熱される。この加熱ゾーンを１０で示す。加熱温度が略液相線ではマグネシウム合金原料３は溶融状態となるが、液相線以下の温度では図示したように固相と液相が混在した半溶融状態となる。また、半溶融状態にあるマグネシウム合金はスクリューの回転撹拌により、図示のようにその剪断力が固相を細かく分断する。ここで、後方の高速射出機構５でスクリュー２を前方に押し出すと、半溶融状態で細かく固相が細断された溶湯が図示のようにノズル９より高速射出され、金型６内に充填されることになる。ここで、凝固まで金型内を加圧保持し、凝固後型を開き成形製品を取り出す。

【００２０】実施例１〜７及び比較例１〜５
低周波炉に鉄ルツボを設置し、ＳＦ$_6$ガス１％（残はドライエア）を溶湯表面に流動させながら実施例および比較例の成分の合金を溶製した。これらの合金を板上に鋳造し、フライス加工にて３〜５ｍｍ径のペレットを製造し、これらを原料として上記成形機を用いて、半溶融成形を行った。

【００２１】

【表１】

		化学組成 （重量%）					
		Ａｌ	Ｃａ	Ｓｉ	Ｍｎ	Ｓｒ	Ｍｇ
実施例１	Ｍｇ-３Ａｌ-２Ｃａ	２.９８	２.０５	０.３０	０.２５	−	残部
実施例２	Ｍｇ-４Ａｌ-２Ｃａ	３.９５	２.０２	０.３０	０.３２	−	↑
実施例３	Ｍｇ-４Ａｌ-３Ｃａ	４.０２	３.０６	０.２５	０.２８	−	↑
実施例４	Ｍｇ-６Ａｌ-３Ｃａ	５.９７	３.１０	０.２８	０.３０	−	↑
実施例５	Ｍｇ-４Ａｌ-２Ｃａ-０.０３Ｓｒ	３.８７	２.０６	０.２５	０.２５	０.０３	↑
実施例６	Ｍｇ-４Ａｌ-２Ｃａ-０.０９Ｓｒ	４.０２	１.９８	０.３０	０.２３	０.０９	↑
実施例７	Ｍｇ-４Ａｌ-２Ｃａ-０.１５Ｓｒ	４.０５	２.１０	０.２３	０.２５	０.１５	↑
比較例１	ＡＳＴＭ　ＡＳ４１相当	４.３９	−	０.４５	０.２８	−	↑
比較例２	Ｍｇ-９Ａｌ-０.５Ｃａ	８.７０	０.４９	０.９０	０.２１	−	↑
比較例３	ＡＳＴＭ　ＡＺ９１Ｄ相当	８.８４	−	０.０２	０.２２	−	↑
比較例４	Ｍｇ-４Ａｌ-４Ｃａ	４.０２	３.９６	０.３２	０.３２	−	↑
比較例５	Ｍｇ-３Ａｌ-３Ｃａ	２.７５	２.７１	０.２７	０.３６	−	↑

【００２２】半溶融成形は型締め力４５０ｔのマシンを用い、その条件は共に射出速度は金型ゲート部において５０ｍ／ｓ、射出圧力約７００ｋｇ／ｃｍ^{2}であり、ノズル部の合金の温度を液相線以下の温度５５０〜５８０℃に設定した。以上の成形条件にて、引張試験片（ＪＩＳ４号試験片）を作成し、ＪＩＳ　Ｚ　２２７１に基づく引張クリープ試験方法により１５０℃、５０ＭＰａでのクリープ特性を検討した。結果を図２に示す。本発明に係るマグネシウム合金は比較例３のＡＺ９１Ｄより耐クリープ性に優れるとされたＡＳ４１より耐クリープ特性に優れることが分かる。

【００２３】また、インストロン引張試験機によりクロスヘッド速度１０ｍｍ／分、測定温度２５℃で破断強度と破断伸びを測定した。結果を表２に示す。アルミニウムが本発明の範囲２〜６重量を越える比較例２、アルミニウム及びカルシウムは本発明の範囲にあるが、Ｃａ／Ａｌ比が０．８を越える比較例４に対し、アルミニウム２〜６重量%、カルシウム０．５〜４重量%を含有し、Ｃａ／Ａｌ比が０．８以下の実施例は優れた伸びを示すことが分かる。

【００２４】

【表２】

	Ａｌ量（重量%）	Ｃａ量（重量%）	伸び（%）
実施例２	３．９５	２．０２	６．７
実施例３	４．０２	３．０６	７．０
実施例４	５．９７	３．１０	５．２
比較例２	８．７０	０．４９	０．８
比較例４	４．０２	３．９６	１．２

【００２５】そこで、実施例と比較例において、Ｃａ／Ａｌ比率と上記伸びとの関係を図３に図示した。これからＣａ／Ａｌ比が０．８を越えると伸びが急激に低下することが分かる。そこで、Ｃａ／Ａｌ比と最小クリープ歪速度との関係をみると、図６に示すように、Ｃａ／Ａｌ比が０．６以下の場合（実施例２）にはより小さなクリープ歪速度となり、更に耐クリープ特性に優れることが分かる。

【００２６】また、図４に示す試験用金型を用い、図示の湯流れを確保して半溶融成形を行うと、表３に示す結果が得られた。これよりＣａ／Ａｌ比率が１に近づくと鋳造割れが円筒部上端オーバーフロー側に生ずるが、Ｃａ／Ａｌが０．８以下ではこのような鋳造割れは一切発生しなかった。

【００２７】

【表３】

	Ｃａ／Ａｌ重量比	鋳造割れの有無
実施例１	０．６９	無し
実施例２	０．５１	無し
実施例３	０．７６	無し
実施例４	０．５２	無し
比較例１	０．９９	有り
比較例５	０．９９	有り

【００２８】一般に鋳造時の滞留時間が長くなると、固相径が急激に増大する（図５の実施例２）が、ストロンチウムを添加しておくと、結晶微細化効果が働き、滞留時間による固相径の増大を抑制することができることが分かる。

【００２９】実施例２の合金素材を用い、図７に示す湯流れ性評価用金型に半溶融成形温度を変化させて溶湯中の固相率を変化させ、図示の方向に溶湯を侵入させ、その湯流れ性を評価した。結果を図８に示す。この結果より固相率３０％を越えると流動長が急激に降下する。この湯流れは成形部材の組織結晶粒径に影響を与えるので、半溶融成形法では固相率３０％以下の状態で成形するのが好ましいことが分かる。

【００３０】半溶融成形ではマグネシウム合金素材を金属粒またはペレットの形態にして使用するが、この金属粒は内部に切削加工などで加工歪を与えておくと、加熱後しばらくした後再結晶粒の核を生成し、固相径を増大していくので、加工歪のない金属粒を用いる場合と加工歪を有する金属粒を比較すると、図１０に示すように固相の成長速度が異なり、成形部材の結晶粒径の微細化には後者のほうが優れていることが理解できる。

【００３１】

【発明の効果】以上の説明で明らかなように、本発明によれば、Ｍｇ－Ａｌ－Ｃａ系耐熱マグネシウム合金部材においてＣａ／Ａｌ比を制御して高温における耐クリープ特性に優れる成形部材を得ることができるので、クラッチピストンおよびクラッチドラムなどの自動車用トランスミッション部品およびロッカーアームなどのエンジン部品を軽量マグネシウム合金で製造して十分な耐久性を持たせることができる。また、本発明では、液相線以下の温度で半溶融成形することにより、従来ダイキャスト法で熱間割れや金型への焼付きの課題を解決しつつ、従来法と同等またはそれ以上の常温および高温強度並びに伸びを保持することができる。

【図面の簡単な説明】

【図１】本発明に係る半溶融成形法及び射出成形法に用いられる成形機の構成を示す概要図。

【図２】　各種マグネシウム合金成形部材のクリープ特性を比較するためのグラフである。

【図３】　各種マグネシウム合金成形部材のＣａ／Ａｌ比と室温伸びの関係を示すグラフである。

【図４】　鋳造割れ試験用金型を示す概略図である。

【図５】　固相径と滞留時間との関係を示すグラフである。

【図６】　各種マグネシウム合金成形部材の最小クリープ歪速度を示すグラフである。

【図７】　各種マグネシウム合金の湯流れ性評価用金型を示す概要図である。

【図８】　図７の金型を使用して測定した実施例２の合金組成における固相率と流動長との関係を示すグラフである。

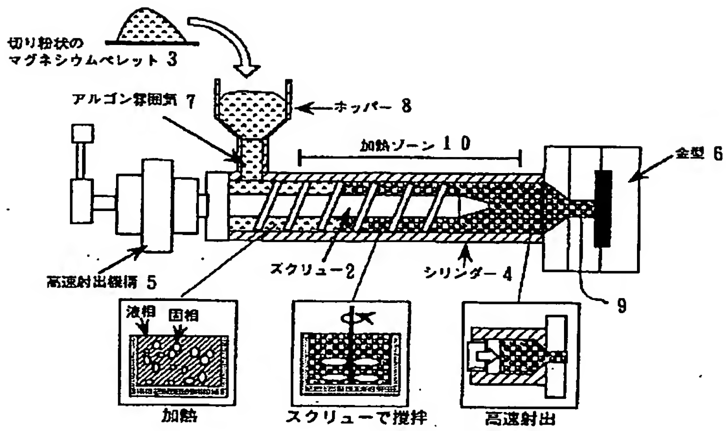
【図９】　実施例３の合金組成から成形された部材の平均結晶粒径と引張強度との関係を示すグラフである。

【図１０】　加工歪のない金属粒と加工歪のある金属粒を使用した場合の固相成長段階を示す模式図である。

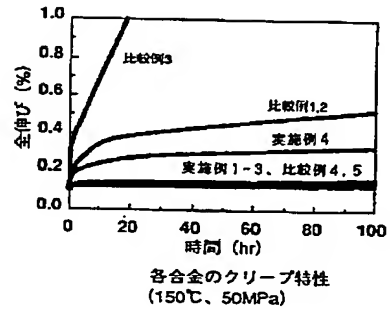
【符号の説明】

１…射出成形機
２…スクリュー
３…原料ペレット
４…シリンダー
５…高速射出機構
６…金型
７…シリンダーへの材料通入路
８…ホッパ
９…ノズル
１０…加熱ゾーン

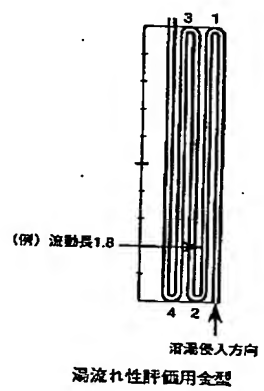
【図1】



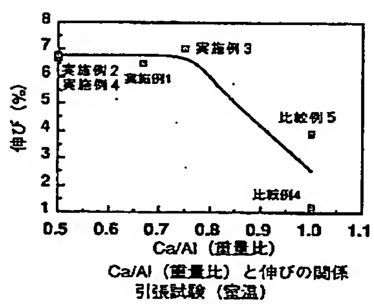
【図2】



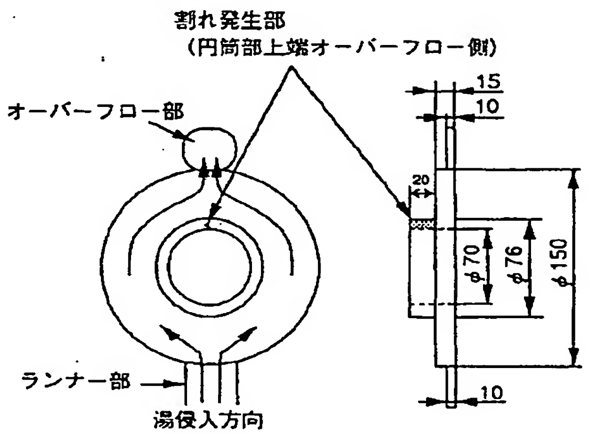
【図7】



【図3】

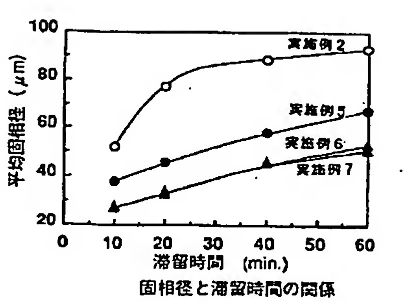


【図4】

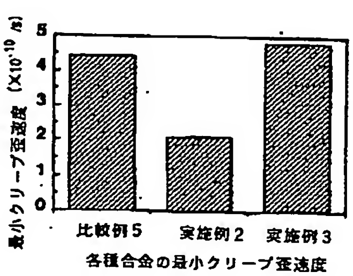


鋳造割れ試験用金型

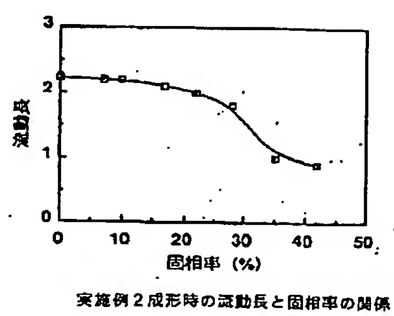
【図5】



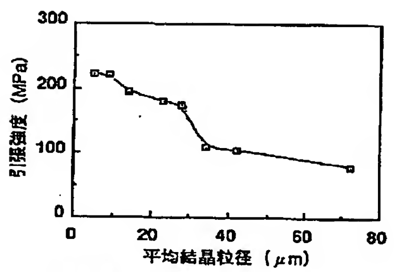
【図6】



【図8】

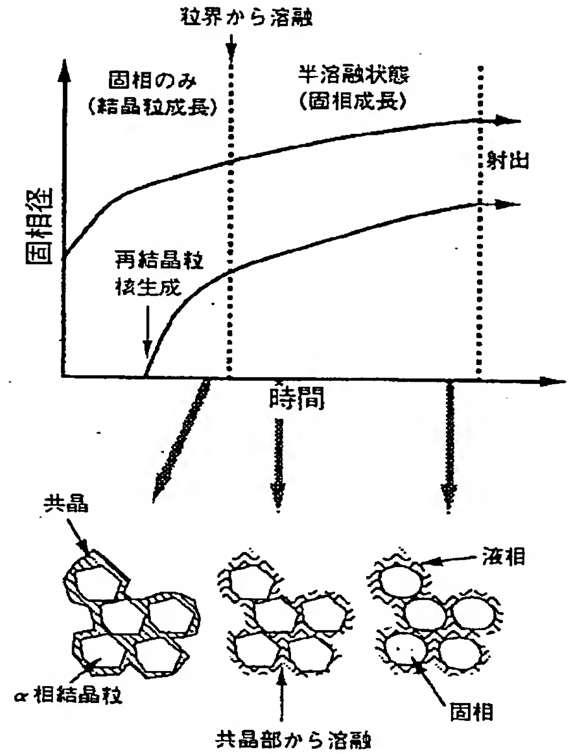


【図9】



実施例3の引張強度と平均結晶粒径の関係

【図10】



固相の成長模式図

フロントページの続き

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